IS 10627 (1983): Methods for Sampling of Pesticidal Formulations [FAD 1: Pesticides and Pesticides Residue Analysis]

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IS : 10627 - 1983
(Reaffirmed 2004)

Indian Standard

METHODS FOR SAMPLING OF PESTICIDAL FORMULATIONS

UDC 632·951 : 543·05

INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

October 1983

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Indian Standard

METHODS FOR SAMPLING OF PESTICIDAL FORMULATIONS

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Indian Standard

METHODS FOR SAMPLING OF PESTICIDAL FORMULATIONS

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 16 June 1983, after the draft finalized by the Sampling Methods for Food Products and Agricultural Inputs Sectional Committee had been approved by the Agricultural and Food Products Division Council.

0.2 Pesticidal formulations are widely used for control of agricultural pests. Keeping in view the important role of agriculture in the growth of Indian economy, it is essential that the pesticidal formulations of desired quality are made available to the users. In order to enable the users to assess the quality of material offered to them, this standard provides the lot acceptance plan. It also includes the provisions of process control for the guidance of manufacturers so as to enable them to control the quality fluctuations and achieve in-built quality of the product.

0.3 The lot acceptance plans for various formulations were earlier included in individual material specifications. However, on the recommendation of the Pest Control Sectional Committee, AFCDC 6, this separate standard for the sampling of pesticidal formulations has been prepared.

0.4 In reporting the results of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS :2-1960*.

1. SCOPE

1.1 This standard prescribes the methods for sampling of pesticidal formulations in solid form (dusting powders, dispersible powders and granules) as well as in liquid form (emulsifiable concentrates and water soluble concentrates).

*Rules for rounding off numerical values (*revised*).

2. GENERAL REQUIREMENTS OF SAMPLING

2.0 In drawing, preparing, storing and handling test samples, the following precautions and directions shall be observed.

2.1 Samples shall be neither taken nor stored at a place exposed to such weather as would effect basic change in its characteristics.

2.2 Precautions shall be taken to protect the samples, the sampling instrument and the containers for samples from adventitious contamination.

2.3 The sampling instrument shall be made of glass or a metal on which the material has no action and shall be clean and dry when used.

2.4 The sample containers shall be of such a size that they are almost but not completely filled by the sample. They shall be clean, dry, leakproof and be made of the material which shall not react with the contents of the sample.

2.5 Once the container is opened, care shall be taken to avoid the risk of deterioration of contents due to the presence of various factors, such as air, light and moisture.

2.6 Each sample container shall be sealed airtight after filling and marked with full details of sample such as date of manufacture, date of sampling, name of the manufacturer and other particulars of the consignment.

2.6.1 The sample or its container shall be marked to indicate the nature of the material and the risks associated with it, wherever necessary.

2.7 The sampler should always be alert for possible biases arising from the use of a particular sampling device or from the segregation of material.

2.8 Before drawing the test samples, contents of each container selected for sampling shall be, as far as possible, thoroughly mixed by suitable means.

2.9 In the case of liquid formulations the proper selection of a representative sample involves a consideration of the physical laws of liquids, their chemical activity, the miscibility of all constituents and the interference of any insoluble materials carried by the liquid.

2.10 Avoid ignition of the material from open flames or sparks produced by static electricity or by metal equipment or tools.

2.11 The operator shall have safe access to and from the place where the sample is to be taken. He shall also have a safe working place wi th necessary light and ventilation.

2.12 The person taking the sample shall be made fully aware of the nature of the hazards involved and precautions to be taken including those prescribed in IS : 4015 (Part 1)-1967* and IS : 4015 (Part 2)-1969†. In the case of toxic nature of material, he should be instructed that in the event of his feeling unwell he should immediately report to the concerned authorities for medical attention.

2.13 The operator engaged in sampling should have clean hands. It may be essential for the operator to wear clean gloves to safeguard against health or other hazards.

2.14 In the case of those formulations which change shape according to temperature, the material may have to be heated to a suitable temperature to bring it into the liquid state from the solid or cooled to a suitable temperature to bring it to solid crystalline state.

3. SAMPING IMPLEMENTS

3.0 The following sampling implements are recommended.

3.1 Formulations in Solid Form — Generally a scoop is recommended.

3.1.1 *Design and Construction of Scoop* — General design of common scoops is shown in Fig. 1. These are normally made of mild steel, but in special cases where the material reacts with steel, the scoop made of any resistant steel (for example, stainless steel) or plastic can be used.

3.1.2 *Procedure* — Dip a scoop of suitable size into the material at an angle, preferably diagonal (but not vertical), at the required spot, withdraw and level off the material with a levelling rod so that the material is not above the sides of the scoop. Transfer the sample drawn into a suitable sample container.

3.2 Formulations in Liquid Form — Generally a sampling tube is recommended.

3.2.1 The sampling tube shall be made of thick glass or of a metal on which material has no action. It shall be of 20 to 40 mm diameter and 400 to 800 mm in length. The glass tube may further be graduated at 10 mm interval. The upper and lower ends are conical and of 5 to 10 mm diameter at the narrow ends. Handling is facilitated by two rings at the upper end. For taking a sample the tube is first closed at the top with the thumb or a stopper and lowered until the desired

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***Guide for handling cases of pesticide poisoning: Part 1 First-aid measures.**

†Guide for handling cases of pesticide poisoning: Part 2 Symptoms, diagnosis and treatment.

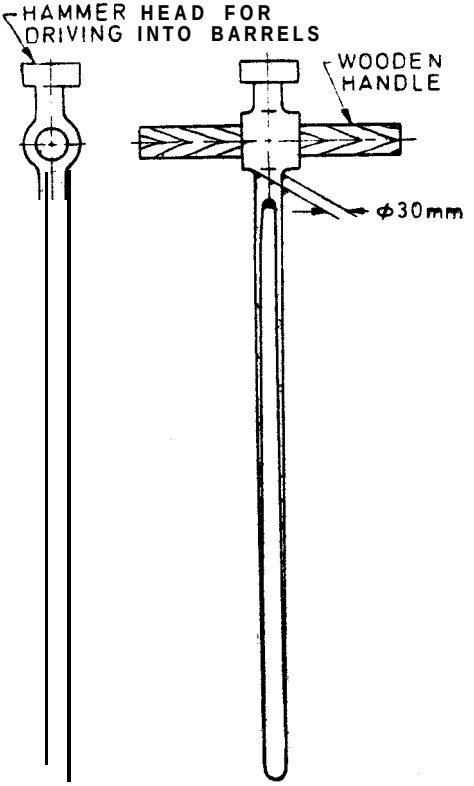


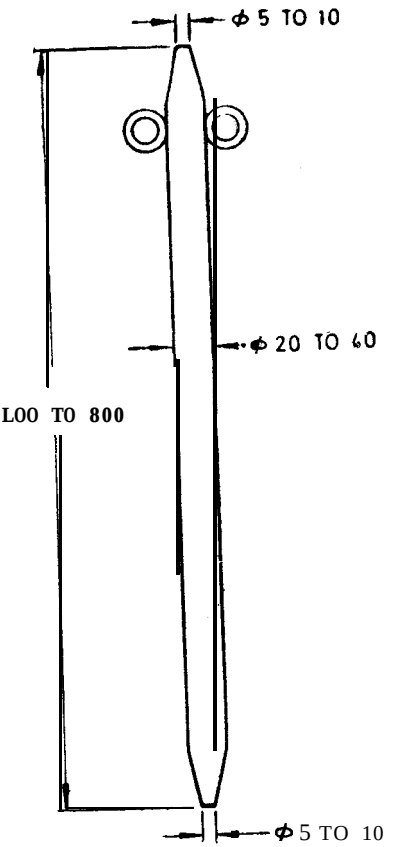
FIG. 1 SAMPLING SCOOP

depth is reached. It is then opened for a short time to admit the material and finally closed and withdrawn. The sampling tube is shown in Fig. 2.

3.2.2 For small containers, a smaller sampling tube of suitable dimensions may be used.

4. PROCESS CONTROL

4.1 In order *to* ensure the desired quality of finished product, appropriate checks shall be made at raw materials and intermediate stages of the process of manufacture.



All dlmensions in millimetres.

FIG. 2 OPEN SAMPLING TUBE

4.1.1 Each consignment of raw materials received in the factory shall be tested on the basis of a representative sample for technical content, moisture and acidity/alkalinity requirements. Alternately, a test certificate shall be obtained from the supplier that the material conforms to the specified requirements. In case the relevant Indian Standard is available, the requirements given there may be referred.

4.1.2 Appropriate checks shall be carried out on the bias of weighing machine, time of mixing various ingredients and performance of the stirrer used for mixing in the case pesticidal formulations in liquid form.

4.1.3 *Formulations in Solid Form* — The process of manufacture may be a batch or a continuous one. *In* a batch process various raw materials are mixed at a time and fed to a grinder or a pulverizer from where the same are conveyed for homogenizing to a blender of known capacity. No further feeding of grinder or pulverizer is possible till such *time* the entire ground material is homogenized in the blender and the same is emptied out. For a continuous process of manufacture various raw materials are continuously fed to the pulverizer from where the same are continuously homogenized in a blender which is continuous to the grinding or the pulverizing unit. In this case there is no pause in the continuity of feeding the pulverizer and the finished material coming out of the blender for final packing.

4.1.3.1 *Batch process* — The quantity of material obtained by mixing various ingredients in a single operation in a mixer, not exceeding one tonne shall constitute a control unit (usually known as a batch in industry). Two representative samples shall be tested from each control unit, before packing, for active ingredient, sieving (in the case of dusting powders) and suspensibility (*in* the case of water dispersible powders) requirements. For remaining requirements, a composite sample prepared by mixing portions of material drawn at regular intervals shall be tested.

4.1.3.2 *Continuous process* — Five tonnes or part thereof, not exceeding a day's production, shall constitute a control unit. Five representative samples shall be taken at regular intervals from each control unit before packing, and tested for active ingredient. Two samples shall be tested for sieving and suspensibility (only in the case of water dispersible powders). The remaining requirements shall be tested on the composite sample.

4.1.4 *Formulations in Liquid Form* — The quantity of material obtained by mixing various ingredients in a single operation in a blending tank shall constitute a control unit. A representative sample shall be tested, before heat stability, for all the requirements. Another representative sample shall be tested, after heat stability, for requirements stipulated in the relevant specification.

4.1.4.1 Before taking the sample, material in the blending tank shall be thoroughly mixed by a suitable stirrer, preferably a mechanical one.

5. LOT INSPECTION

5.1 If the manufacturer has maintained an adequate and satisfactory system of quality control in the manufacture of pesticidal formulations, the resulting data and information may be made available to the purchaser along with the material supplied to enable him to judge the acceptability of the consignment. When it is not possible to provide

this information or if the purchaser so desires, the procedure laid down in the following clauses shall be followed for determining the conformity of the material to the requirements of the specification.

5.2 **Scale of Sampling**

5.2.1 *Lot* — In a single consignment, all the containers of the same type, same grade and belonging to the same batch of manufacture shall be grouped together to constitute a lot.

NOTE — **The definition of batch shall depend on the type of process of manufacture, that is, whether a batch process or a continuous one. The distinction between the two processes is explained in 4.1.3.**

5.2.2 For ascertaining the conformity of the material to the requirements of the individual specification, samples shall be tested from each lot separately.

5.2.3 *Formulations in Solid Form*

5.2.3.1 The number of containers to be chosen from a lot shall depend on the size of the lot and shall be according to Table 1.

TABLE 1 NUMBER OF CONTAINERS TO BE CHOSEN FOR SAMPLING

QUANTITY OF MATERIAL IN TEE LOT (kg)	SAMPLE SIZE (NUMBER OF CONTAINERS)	
	For Containers of Up to 25 kg	For containers of More than 25 kg
(1)	(2)	(3)
up to 1 000	3	2
1 001 to 3 000	5	3
3 001 to 10 000	7	5
10 001 and above	10	7

5.2.4 Formulations in Liquid Form

5.2.4.1 Three container shall be chosen from a lot if the material is packed in containers of up to 5 litres and two containers if the material is packed in containers of more than 5 litres, irrespective of the size of the lot.

5.2.5 The containers required for sampling according to 5.2.3 and 5.2.4 shall be selected at random. In order to ensure the randomness of selection, either of the following procedures may be followed.

5.2.5.1 *Simple random sampling* — **In case** the lot consists of containers such that each container is easily identifiable, this method may be followed while selecting the containers for sampling. According to this method,

the sample of thk requisite size n is drawn from the lot of size N, in such a manner that, while selecting a container, the chance for any container of the lot being included in the sample is the same. For the selection of a simple random sample of n containers from the lot of N containers, the first requisite is to obtain n random numbers which lie in the range of 1 to N. For this purpose starting from any number of the random number table (see Appendix B of IS :4905-1968*) and continuing on with the numerals, in any direction, right or left, up or down, the succeeding numerals are copied out one by one till n different numerals are obtained. The numerals zero or those which are greater than Nor those which have already occurred, shall be omitted. The numerals noted down in this manner shall then be arranged in the ascending order of magnitude. Starting from any container in the lot and counting them in one order, the containers corresponding to the numerals already noted down shall be withdrawn to constitute the required sample of size n.

Example 1 — It is desired to obtain a sample of 5 containers each of 25 kg from a lot of ‘2 500 kg (100 containers each of 25 kg). If the containers in the lot are mentally assigned the serial numbers up to 100, the problem then is to obtain 5 random numbers in the range of 1 to 100. Taking the 2-digited random numbers from Appendix B of IS : 4905-1968* and starting from any number say 64 occurring in second row and 11 and 12 columns on page 19 of IS :4905-1968* and proceeding downwards, the numerals less than 100 are noted down. Thus the 5 numerals so obtained are 19, 09, 80, 34 and 45. When arranged in ascending order of magnitude, the numerals become 9, 19, 34, 45 and 80. The containers in the lot corresponding to these numbers shall then be selected to constitute the required sample of size 5.

5.2.5.2 *Systematic sampling* — When the containers in the lot are presented in an orderly manner, it is possible to considerably simplify the selection of the random sample of the required size. Instead of choosing the desired number of random numerals and then drawing the containers corresponding to these numerals, one container is chosen at random from the lot and thereafter the containers are selected regularly at predetermined intervals. The method consists of first selecting *a single* sample container from the population of N containers and thereafter selecting containers at regular intervals to make up the desired sample of size n. For this purpose, the integral part of N/n (say r) is taken as the interval and then the containers are counted in one order and every tth container thus counted is withdrawn until the sample of required size is obtained.

Example 2 — It is desired to take a random sample of 3 containers each of 25 kg from a lot of 1 000 kg that is 40 containers each of 25 kg, according to Table 1. For this purpose, if the containers in the lot are

*Methods for random sampling.

stacked so that they can be identified, calculate the integral part of $\frac{40}{3}$, which is 13. Starting from any container at random, count the containers in one order as 1, 2, 3, ... , 13 and so on. Every 13th container so counted shall be chosen till 3 containers are obtained to constitute the desired sample. In this case, the sample containers to be selected are those corresponding to serial numbers 13, 26 and 39.

5.3 Test Samples and Referee Samples

5.3.1 *Formulations in Solid Form* — Before drawing the test samples, mixing the contents of each container by shaking is usually not practical. Draw small portions of the material from as many places as practicable throughout the volume of each selected container (see Table 1). The total quantity of material drawn from each container shall be sufficient so as to make triplicate determinations for all the characteristics given in the individual specification, Mix thoroughly all the portions of material from the same container.

5.3.2 *Formulations in Liquid Form* — Before drawing the test samples, the contents of each container shall be mixed by shaking, agitating, rolling or any other suitable means so as to bring all portions into uniform distribution. A representative portion of material shall be taken from each selected container (see 5.2.4.1). For this purpose, approximately equal quantity of material shall be taken from top, middle and bottom of the container and well mixed to constitute the sample. The quantity of material drawn from each container shall be sufficient so as to make triplicate determinations for all the characteristics given in the individual specification.

5.3.3 A small but approximately equal quantity of material shall be taken from each of the selected containers and shall be well mixed so as to form the composite sample. The quantity of material in the composite sample shall be sufficient so as to make triplicate determinations for the specification requirements to be tested on the composite sample. The composite sample shall be divided into three equal parts, one for the purchaser, another for the vendor and the third for the referee.

5.3.4 The remaining portions of the material (after the quantity needed for the formation of the composite sample has been taken) shall be divided into three equal parts. These parts shall be immediately transferred to thoroughly dried sample containers which are then sealed air-tight and labelled with all the particulars of sampling. For this purpose suitable containers as given in IS : 8190 (Part 1)-1976* for formulations in solid form and in IS : 8190 (Part 2)-1976† for formulations in liquid form may be used. The material in each sealed sample

*Requirements for packing of pesticides: Part 1 Soild pesticides (*first revision*).
†Requirements for packing of pesticides: Part 2 Liquid pesticides (*first* revision).

container shall constitute an individual test sample. These individual test samples shall be separated into three identical sets of test samples in such a way that each set has a sample representing each selected container. One of these three sets shall be marked for the purchaser, another for the vendor and the third for the referee.

5.3.5 ***Referee Sample*** — Referee sample shall consist of a composite test sample and a set of individual test samples marked for this purpose and shall bear the seals of the purchaser and the vendor. These shall be kept in a cool and dry place as agreed to between the purchaser and the vendor so as to be used in case of dispute between the two.

5.4 Number of Tests

5.4.1 ***Formulations in Solid Form*** — Active ingredient, sieving (only for dusting powders) and suspensibility (only for water dispersible powders) shall be tested on individual samples and the remaining requirements on the composite sample.

5.4.2 ***Formulations in Liquid Form*** — Active ingredient and emulsion stability shall be tested on individual samples and the remaining requirements on the composite sample.

5.5 Criteria for Conformity

5.5.1 The lot shall be declared as conforming to the requirements of the specification if 5.5.1.1, 5.5.1.2 and 5.5.1.3 are satisfied.

5.5.1.1 All the test results for each of the characteristics tested on individual samples (see 5.4.1 and 5.4.2) satisfy the relevant specification requirements.

5.5.1.2 The average of all the test results for active ingredient is greater than or equal to the nominal value.

5.5.1.3 For the remaining requirements (see 5.4.1 and 5.4.2), all the test results on the composite sample satisfy the relevant specification requirements.

AMENDMENT NO. 1 JULY 1985

TO

IS : 10627-1983 METHODS FOR SAMPLING OF PESTICIDAL FORMULATIONS

Alterations

(Page 5, *clause* 3.1) — Substitute existing clause 3.1 by the following:

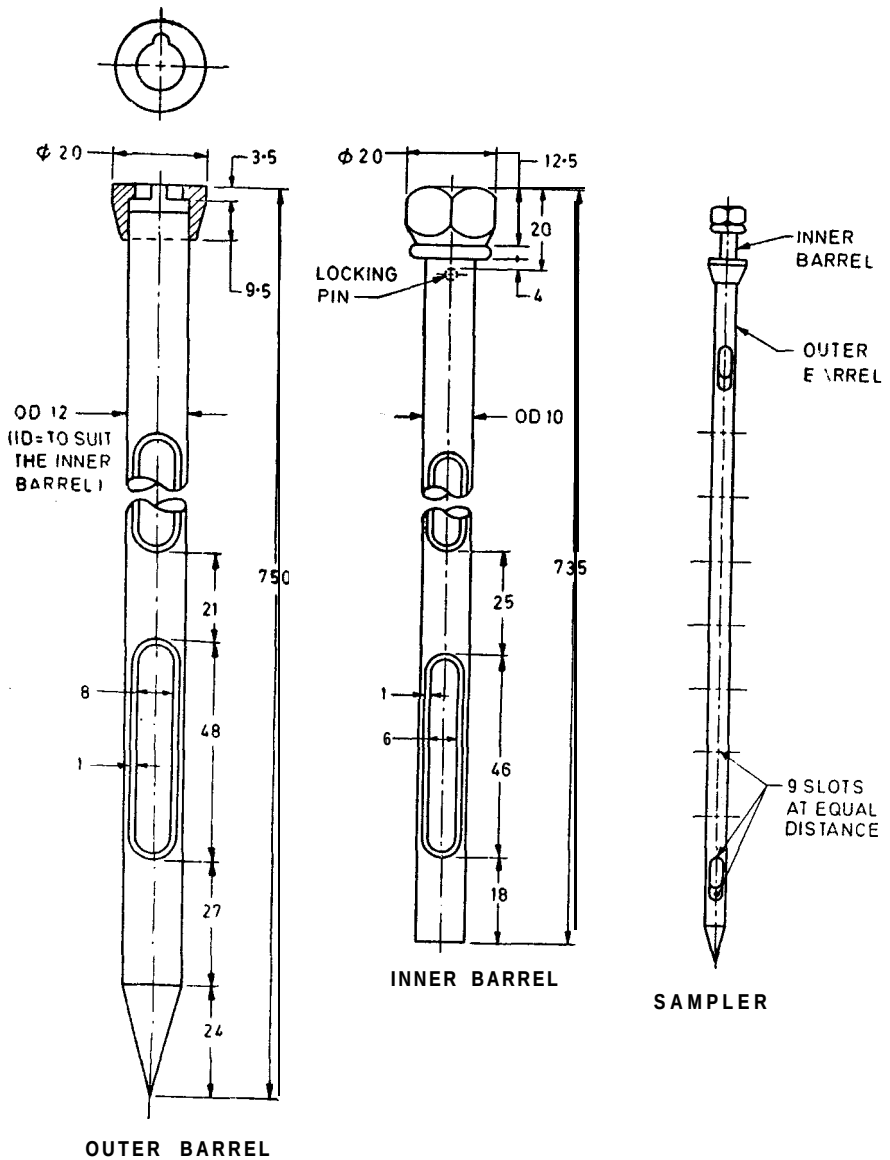
'3.1 Formulations in Solid Form — Generally a sampler (or bag trier) consisting of two barrels (outer and inner) having slots at equal intervals, as shown in Fig. 1, is recommended. The working end of the outer barrel is pointed for easy insertion into the bag. The sampler shall be made of steel, brass or any other suitable material for taking the material from different layers of the bag. The slots of the sampler can be opened or closed by rotating the inner-barrel-head. The sampler is inserted into the bag in a closed position. After inserting the sampler to the desired depth, the sample material is collected by opening the slots of the sampler and then closing them. The sampler after this shall be taken out from the bag and then emptied on a smooth flat surface table/paper/any other suitable container. Thus the sample of requisite quantity is collected and difference in the material taken out from different layers of the bag can also be studied.

NOTE — The tops of the inner and the outer barrels should be convenient for handling.'

(*Page* 6, *Fig.* 1) — Substitute the existing Fig. 1 by the figure appearing on page 2.

(*Page* 6, *clause* 4) — Insert the following new clause under this clause:

'4.0 Following process control provisions are recommended for the guidance of manufacturer.'



All dimensions in millimetres.

FIG. 1 TYPICAL SAMPLER FOR FORMULATIONS IN SOLID FORM (**AFDC** 57)

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